



California Environmental Protection Agency

AIR RESOURCES BOARD

Monitoring and Laboratory Division
Air Quality Surveillance Branch

Protocol for the Application Air Monitoring of Propyzamide

August 31, 2009

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The following protocol has been reviewed and approved by staff of the Air Resources Board (ARB). Approval of this protocol does not necessarily reflect the views and policies of the ARB, nor does the mention of trade names or commercial products constitute endorsement or recommendation for use.

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1.0 Introduction

At the request of the California Department of Pesticide Regulation (DPR), (January 4, 2008 Memorandum, Warmerdam to Goldstene) the Air Resources Board (ARB) staff will determine airborne concentrations of propyzamide in Monterey County. Ambient air sampling will occur prior to, during and after an application of propyzamide. This monitoring will be performed to fulfill the requirements of AB 1807/3219 (Food and Agricultural Code, Division 7, Chapter 3, Article 1.5, Section 14022(c)) which requires the ARB "to document the level of airborne emissions.... of pesticides which may be determined to pose a present or potential hazard..." when requested by the DPR. Monitoring is being conducted to coincide with the use of propyzamide as a selective herbicide on leaf and head lettuce for human consumption.

The study will consist of six (6) sampling periods: A background sample period, an application sampling period, a post application sampling period ending one (1) hour before sunset, two (2) overnight sampling periods and two (2) daytime sampling periods. The application sampling period will begin thirty minutes prior to the application of propyzamide. There will be a total of 58 sample XAD resin sorbent tubes (4 background, 6 field spikes, 1 trip spike, 1 trip blank, 6 collocated, 32 application and 10 spares).

Background sampling will begin one (1) to seven (7) days prior to the start of the propyzamide application and will sample for a minimum of 12 to 24 hours. Background sampling will consist of four (4) background samples, one (1) collocated sample and one (1) collocated sampler loaded with a field spike.

2.0 Chemical Properties of Propyzamide

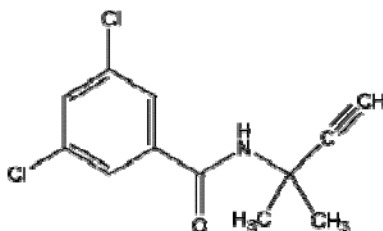


Figure 1
Chemical structure of Propyzamide

Propyzamide is an odorless white or off-white crystalline solid (Kidd and James 1991). It is relatively stable and there should be little decomposition of this material (Berg 1986). Propyzamide is noncorrosive. It poses a fire hazard if exposed to heat or flame and explosion hazard in the presence of strong oxidizers. Thermal decomposition of propyzamide will release toxic oxides of nitrogen and carbon and toxic and corrosive fumes of chlorides (OHS 1991). It is soluble in water (15 mg/L at 25 °C), methanol (150 g/L), isopropanol (150 g/L), cyclohexanone (200 g/L), methyl ethyl ketone (200 g/L) and dimethyl sulfoxide

(330 g/L), moderately soluble in benzene, xylene and carbon tetrachloride and slightly soluble in petroleum ether (BCPC 2000). Table 1 lists some of its physical and chemical properties.

Chemical name	Propyzamide
Common name	Propyzamide, pronamide
Registered Trade names or other names [†]	Clanex, Kerb, Judo, Piza, RH-315 Rapier, Ronamid
CAS number	23950-58-5
CDPR Chemical Code	694
Empirical formula	C ₁₂ H ₁₁ Cl ₂ NO
Molecular weight	256.1
Appearance	Colorless, odorless powder
Vapor pressure	0.058 mPa (at 25 °C) 8.5 x 10 ⁻⁵ mmHg (at 25 °C)
Melting point	155 - 156 °C
Solubility	15 mg/L (ppm) (at 25 °C)
Henry's Law constant	9.8 x 10 ⁻⁹ (at 25 °C)
Octanol-water partition coefficient (log Kow)	3.1 – 3.2
Soil adsorption coefficient (Koc)	889 cm ³ /g
Hydrolysis half-life (20 °C)	42 days
Soil dissipation	53.3 days (17 °C) 53.7 days (23 °C)
Soil degradation half-life	392 days (aerobic conditions, 26 °C) 762 days (anaerobic conditions, 26 °C)
Stability	Decomposes above m.p., degraded photolytically on soil thin films, DT ₅₀ 13-57 days in artificial sunlight. In solution for 28 days at 20 °C < 10% loss at pH 5-9
Breakdown products	3,5-dichlorobenzamide
Sources: CDPR; BCPC 2000; EXTTOXNET [†] <u>Disclaimer:</u> The mention of commercial products, their source, or use in connection with material reported herein is not to be construed as either an actual or implied endorsement of such products.	

Table 1
Physical and chemical properties of Propyzamide

3.0 Project Goals and Objectives

The goal of this monitoring project is monitoring to measure the concentrations of propyzamide in the ambient air prior to, during, and after a field application.

To achieve the project goal, the following objectives should be met:

1. Identification of monitoring sites that mutually satisfies criteria for application air sampling and DPR's requirements.
2. Appropriate application of sampling/monitoring equipment to determine application propyzamide concentrations.
3. Application of relevant field quality assurance/quality control practices to ensure the integrity of field samples.
4. At the conclusion of the project, MLD will provide DPR with a final report with all relevant information and data of this project.

4.0 Contacts

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5.0 Study Location

The propyzamide application is planned for August 19, 2009 in King City, California which is located in Monterey County.

6.0 Study Design

The propyzamide sampling events, times and duration are listed below. For August 2009 the approximate sunrise time is 0445 PST and the approximately sunset time is 1930 PST.

The proposed sampling events are subject to change due to state work force furloughs and overtime policies. The post application sampling may be reduced or lengthened.

Sampling Events, Times and Durations are as follows:

- **Event 1:** Background sampling minimum 12 to 24 hours prior to application (Tuesday 18th).
- **Event 2:** Application sampling start time is approximately 30 minutes prior to application. Sampling will end one (1) hour after completion of application (Wednesday 19th).
- **Event 3:** Post application sampling starts one (1) hour after application and ends one (1) hour before sunset (Wednesday 19th).
- **Event 4:** Post application sampling overnight starts one (1) hour before sunset and ends one (1) hour after sunrise (Thursday 20th).
- **Event 5:** Post application sampling starts one (1) hour after sunrise and ends one (1) before sunset (Thursday 20th).
- **Event 6:** Post application sampling overnight one (1) hour before sunset and removed one (1) hour after sunrise (Friday 21st).

ARB personnel contacts Rio Farms representative to inform them that air sampling has ended and all ARB personnel and equipment have been removed.

Details of Sampling Events:

- a) Background sampling will start the day before the application with minimum sample duration of twelve hours, but no more than twenty four hours. The background samples will be removed prior to the start of the application. The background samplers will be located within 20 meters from the lettuce field which is going to be treated. The field spike will be spiked with a concentration of 3.0 ug/m^3 of propyzamide.
- b) The propyzamide application sampling will consist of six (6) sampling events including the background. The application sampling event will start approximately thirty minutes prior to the start of propyzamide application. Sampling will continue until one (1) hour after the application is completed. A post application sampling period will immediately start and then end one (1) hour before sunset. The rest of the study will consist of two (2) overnight sampling events and two (2) daytime sampling events. Each overnight sampling event will start one (1) hour prior to sunset and end one (1) hour after sunrise. Each daytime sampling event will start one (1) hour after sunrise and end one (1) hour before sunset.
- c) There will be eight (8) sampling sites around the lettuce field. For a square field, four (4) sites will be located at each corner and four (4) sites will be located midway on each side. The estimated downwind site will have a collocated sample on the regular sampler. All sampler intakes will be 1.7 meters (67 ± 6 inches) above the ground. Samplers will be placed within 20 meters (66 feet) from the drip line of the foliage.
- d) Each air sampler will consist of an ARB Pesticide Stick Sampler (PS sampler), a rotameter to control the flow, rubber tubing to retain the XAD resin sorbent tube in the flow path, a rain/sun screen and a DC pump box with a battery box connected. The PS sampler is powered by 12 VDC gell cell batteries. After each sampling event the used battery will be replaced with a newly charged battery. Flows will be set at 3.0 lpm and the average of the start and stop flows shall be $3.0 \text{ lpm} \pm 20\%$ ($2.4 - 3.6 \text{ lpm}$). Figure 1 (Pesticide Stick Sampler) demonstrates an assembled sampler.

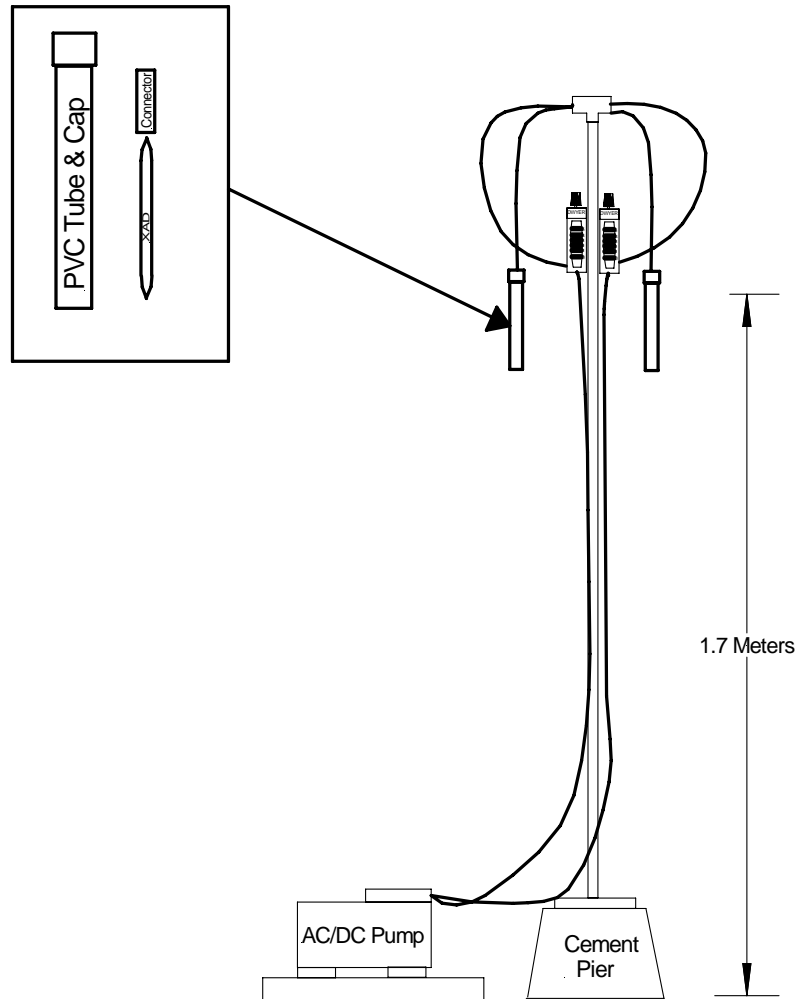


Figure 2
Pesticide Stick Sampler

- e) In order to reduce direct pesticide exposure to ARB staff during the pesticide application event, all samplers will begin a minimum of thirty minutes prior to the start of the application. At the end of each sampling period the following general procedure will occur at each site; Flows will be verified, documentation completed, all XAD resin sorbent tubes will be removed, capped, labeled and placed in a freezer bag which will in turn be placed in a dry ice cooler, the battery replaced, a new XAD resin sorbent tube installed and flows adjusted if necessary. Field notes and observations will be recorded (such as propyzamide application flow rate and total amount of propyzamide applied).

- f) Meteorological data will be collected using a Met One Instruments' AutoMet Digital Meteorological Monitoring System located on a trailer with a crank up tower. The AutoMet will be located no closer than twenty meters from the edge of the field being monitored. The meteorological sensors will be installed between 5.9 and 6.9 meters above the ground. The AutoMet station will continuously measure and record 5 minute averages for wind speed, wind direction, ambient temperature and percent relative humidity throughout the background, application and post application sampling periods.
- g) The MLD will provide DPR with a final report containing all relevant information, collected data and analytical results gathered during the course of the study.

7.0 Sampling and Analysis Procedures

Special Purpose Monitoring Section (SPM) personnel will hand-carry samples to and from MLD's laboratory in Sacramento, and to and from the sampling location. The samples will not be exposed to extreme conditions or subjected to rough handling that might affect sample integrity.

At each sampling site, the operator will assure all required sample collection information is recorded on the affixed XAD resin sorbent tube identification labels and field log sheets. After collection the samples are placed in a glass tube and stored in a cooler at 4° C or less until returned to the laboratory. The sample tubes will be transported on dry ice, as soon as reasonably possible, to the ARB Sacramento Monitoring and Laboratory Division laboratory for analysis. These samples will be stored in the freezer or extracted/analyzed immediately. Samples are collected in the field with a flow rate of 3.0 liters per minute (lpm).

All reported sampling times, including meteorological data, will be reported in Pacific Standard Time (PST).

The Northern Laboratory Branch (NLB) will supply SPM with XAD resin tubes. NLB will perform analyses for propyzamide on collected application samples and report results to SPM.

Laboratory analyses will be performed in accordance with applicable standard operating procedures (Standard Operating Procedure Sampling and Analysis of Propyzamide). The SOP is included in this Protocol as Appendix A.

The following XAD resin tube validation and analytical quality control criteria should be followed during pesticide analysis.

1. **Sample Hold Time:** Sample hold time criteria will be established by the Laboratory. Samples not analyzed within the established holding time will be invalidated by the Laboratory.
2. **Duplicate Analysis:** Laboratory to establish relative percent difference (RPD) criteria for duplicate analysis. Replicate analysis shall be performed on every tenth sample (10%) per analytical batch sequence (excluding standard controls and other laboratory quality control data). Replicate analysis shall be performed on every tenth (10%) per analytical batch sequence (excluding standard controls and other laboratory quality control data). Lab to provide duplicate analytical results and RPD.
3. **Method Detection Limit (MDL):** MDL sample analytical results less than the MDL shall be reported as a less than numerical value. This less than numerical value shall incorporate any dilutions/concentrations.
4. **Analytical Linear Range:** Any analytical result greater than the highest calibration standard shall be reanalyzed within the calibrated linear range.

8.0 List of Field Equipment

<u>Quantity</u>	<u>Item Description</u>
(1)	Met-One Auto met portable meteorology system having calibrated sensors to measuring 5 minute averages for wind speed, direction, ambient temperature, and relative humidity w/built-in data logger.
(1)	Measuring Wheel
(1)	200 ft measuring tape
(1)	Tripod and compass
(1)	Global Positioning System (GPS) with backup batteries and carrying case
(1)	Digital Camera with backup batteries and carrying case
(2)	Alborg mass flow meter 0-5 Lpm.
(12)	Sampling trees/pumps
(68)	XAD resin tubes (4 background, 40 application, 6 collocated, 6 field spikes, 1 trip spike, 1 trip blank, and 10 spares).
(82)	Batteries
(4)	Power Strips
(2)	Extension cords
(1)	Miscellaneous tubing and fittings
(1)	Box of banana plug wires for pump and battery connection
(8)	Battery Chargers

9.0 Quality Control

Quality control procedures will be observed to ensure the integrity of samples collected in the field. National Institute of Standards and Technology (NIST)-traceable transfer standards will be used to calibrate meteorological sensors and measure sample flow rates.

The metrological sensors will be calibrated and aligned following the procedures outlined in the standard operating procedures on the Air Monitoring Web Manual at the following link.

<http://www.arb.ca.gov/aqdas/amwmn.php?c=5&t=sop>

Each XAD resin tube will be assigned a field sample number that provides for identification of site, sample ID number, operator, and sample information as well as sample transfer information.

Field Spike (FS): Six (6) field spikes will be prepared by the laboratory by injecting a XAD resin tube with 3 nanograms (ng) of propyzamide. The field spikes will be installed for each sampling event including the background. and will be located next to the collocated air sampler.

Trip Spike (TS): A trip spike will be prepared by the laboratory by injecting a XAD resin tube at the same level as the field spike. The trip spike will be transported and analyzed along with the field spike. The trip spike is treated the same as a field spike with exception that it is not installed onto a sampler.

Field Blank (FB): A field blank will be a XAD resin tube opened in the field and return but is not installed onto a sampler.

Trip Blank (TB): A trip blank will be an unopened XAD resin tube to the field and return but is not installed onto a sampler.

Collocated (CO): Collocated (side-by-side) air samplers will operate at the predominantly downwind sample site throughout all sampling events.

Site/Sample Identification

The propyzamide application sampling sites will be named accordingly for the background, ambient, application, and post application as follows:

Background Site Naming:

Site-1-BKGD
Site-1CO-BKGD
Site-1-FS

Letter Abbreviations as follows

N = North Side
S = South Side
W = West Side
E = East Side
BKGD = Background Sample
FS = Field Spike
CO= Co-located
NEC = NE Corner Sample
NWC = NW Corner Sample
SEC = SE Corner Sample
SWC = SW Corner Sample
TS = Trip Spike
TB = Trip Blank
FB = Field Blank

Application Site Naming:

Site-1N Site-2NEC
Site-3E Site-4SEC
Site-5S Site-6SWC
Site-7W Site-8NWC

Following the quality control procedures listed above will ensure the quality and integrity of the samples collected in the field and will ensure accurate field and lab data collection.

10.0 Deliverables

10.1 Air Quality Surveillance Branch Deliverables

Within 60 days from receipt of the final results report from the Northern Laboratory Branch (NLB), AQSB will provide DPR with a report containing the following topics:

- 1) Sampling Protocol.
- 2) Personnel Contact List.
- 3) Site Maps.
- 4) Site Photographs.
- 5) Site Descriptions and Measurements
Site, sampler, GPS coordinates, inlet height, distance to roads, site-specific comments, and total pounds of propyzamide applied per acre.
- 6) The distance and direction of the sampler to the treated or potentially treated fields.
- 7) A map of the monitoring site locations.
- 8) Sample Summary Table.
- 9) Field Sample Log.
- 10) Laboratory Analysis Reports with calculations in electronic format.
- 11) Met Station and Sampler Calibration Reports.
- 12) Transfer Standards' Certification Reports.
- 13) Disk containing electronic files of 5-minute averaged Meteorological Data.
- 14) Disk containing electronic files of Report.

Also provided in the application monitoring report:

- 1) An accurate record of the positions of the monitoring equipment with respect to the field, including the exact distance that the sampler is positioned from the field.
- 2) An accurate drawing of the monitoring site showing the precise location of the meteorological equipment, trees, buildings, etc..
- 3) Meteorological data collected at a minimum of 1 minute intervals (averages) including wind speed, wind direction, humidity, air temperature, and comments regarding degree of cloud cover.
- 4) The elevation of each sampling station with respect to the field.
- 5) The orientation of the field with respect to North (identified as either true or magnetic north). Samples collected during fog episodes will be designated as such.

In addition, the Special Purpose Monitoring Section (SPM) will prepare a project binder containing the above information. This binder will remain with SPM though available for viewing and review as requested.

10.2 Northern Laboratory Branch (NLB) Deliverables

Within 60 days from the last day of analysis, The NLB will provide SPM with a report that will include the following topics:

- 1) Analytical result table(s) to include:
 - a. Sample identification (name).
 - b. Date sample received from field.
 - c. Date sample analyzed.
 - d. Dilution ratio (if applicable)
 - e. Analytical results.
 - f. Quality control results.
- 2) Sample equations used in calculating analytical results.
- 3) Table of duplicate/replicate results including calculated relative percent difference (RPD).
- 4) Table of analytical results from all field spikes.
- 5) Table of analytical results from all trip and laboratory spikes including percent recoveries.
- 6) Table of analytical results from all trip blanks.
- 7) Table of analytical results from all laboratory blanks, standards and control checks performed, including dates performed and relative percent recoveries if applicable.
- 8) Copy or location of analytical method or Standard Operating Procedures (SOP) used for analysis.
- 9) Section or provision listing or reporting any and all deviations from analytical SOP and this protocol.

APPENDIX A:
Standard Operating Procedure Analyses for
Propyzamide

The Special Analysis Laboratory Section of MLD's Northern Laboratory Branch will perform the analyses for propyzamide collected by the XAD resin tube method. This analytical procedure is entitled, Standard Operating Procedure Sampling and Analysis of Propyzamide.

California Environmental Protection Agency



Air Resources Board

**Standard Operating Procedure
Sampling and Analysis of Propyzamide**

**Special Analysis Section
Northern Laboratory Branch
Monitoring and Laboratory Division**

June 2009

Version 1

Approved by:

**Russell Grace, Manager
Special Analysis Section**

1. SCOPE

This is a gas chromatography/electron capture detector (GC/ECD) method for the determination of propyzamide in ambient and application air sampling.

2. SUMMARY OF METHOD

Ambient and application air samples are collected on XAD-2 sorbent tubes. Sampled tubes are stored at four (4) degrees centigrade (°C) or lower prior to extraction. Sample tubes are extracted using pesticide grade ethyl acetate. Sample analysis is performed using a GC/ECD. Sample analysis and quantitation uses external standard method for instrument calibration. Estimated quantitation level (EQL) for this method is approximately 5.0 micrograms per cubic meter ($\mu\text{g}/\text{m}^3$) prior to any sample dilution.

3. INTERFERENCES / LIMITATIONS

Method interference may be caused by contaminants in solvents, reagents, glassware and the XAD-2 tubes that can lead to discrete artifacts or elevated baselines. Analysis of samples containing high concentrations of early eluting components may cause significant contamination of the analytical equipment. Both a system blank and method blank must be analyzed with each batch of samples to detect any possible method or instrument interference.

4. EQUIPMENT AND CONDITIONS

A. Instrumentation

- Agilent Technologies 7890 Series gas chromatograph with Agilent Model 7683B injector.
- Column: Agilent HP-5, 30 meter, 0.32mm I.D., 0.25 micron film thickness, with helium as carrier gas at constant flow and nitrogen as the makeup gas at 25 ml/min.
- GC temperature program: initial 100° C, initial time 2 minutes, to 260° C @ 12° C/min, hold 2 minutes.
- Inlet temperature 260° C; splitless.
- Detector temperature 325° C.

B. Auxiliary Apparatus

- XAD-2 tubes (400/200 mg) (SKC cat # 226-30-6) or equivalent
- Glass amber vials, 2-ml capacity with septum caps.

C. Reagents

- Ethyl Acetate(EtAc) (B&J brand HPLC grade or equivalent)
- Propyzamide 98.1%, Chem Services PS-349

5. SAMPLE COLLECTION

- a) Samples are collected in the field with a maximum flow rate of three (3) liters per minute (lpm).
- b) After collection the samples are placed in a glass tube and stored in a cooler at 4° C or less until returned to the laboratory.
- c) Samples are stored at 4° C or less until ready for analysis.

6. SAMPLE EXTRACTON

- a) Prepare a method blank and laboratory control sample (LCS) tube with every batch of field samples not to exceed twenty (20) samples in an analytical batch. The LCS is spiked with 3 µg of propyzamide before extraction.
- b) Carefully score and break the sample XAD-2 tube just in front of the glass wool plug on the primary section.
- c) Remove the glass wool plug using forceps. Pour the XAD-2 resin from the primary section into an 8 ml glass vial.
- d) Score the tube just in front of the secondary section glass wool. Retain the secondary section for later analysis to check for breakthrough.
- e) Using three (3.0) ml of EtAc carefully rinse the inside of the primary section into the glass vial. Cap securely.
- f) The extracts are allowed to sit at room temperature for 1 hour. The extracts are ready for analysis or if not analyzed are stored in a refrigerator at 4° C.

7. ANALYSIS OF SAMPLES

- a) Transfer approximately 0.25 ml of the sample extract into a 1.5-ml autosampler vial equipped with a 0.25 ml insert. Sample extract is now ready for analysis.
- b) A 1-µl injection volume will be used for all analyses.
- c) Perform a calibration curve using concentrations at or near the EQL to approximately 10 times higher. At least five (5) points must be analyzed to establish a calibration curve.

- d) Prepare a sample sequence for the GC/ECD. The sequence should include a system blank and a calibration control standard, for every ten (10) samples analyzed. If this batch of samples includes a method blank and /or LCS, they should be run prior to field samples to verify that QC criteria have been met.
- e) Because of the nature of the XAD-2 tube, extraneous components will be extracted along with the analytes of interest. To minimize excessive carry over of these contaminants from one analysis to the next, a system blank should be run after every ten (10) to twenty (20) samples or more frequently if indicated by sample chromatograms. In no case should a sample contaminant interfere with the peaks of interest. This will be verified by the absence of a peak in the analyte retention time window during the system blank analysis.
- f) Review and edit the quantitation reports as needed.
- g) The samples must be diluted if the analytical results are not within the calibration curve. Every attempt should be made to have the diluted results fall within the upper half of the calibration curve.
- h) The final results will be adjusted by an appropriate dilution factor and reported in µg/ml.
- i) The atmospheric concentration is calculated according to:

$$\text{Ambient Sample Conc. (}\mu\text{g/m}^3\text{)} = \frac{\text{Extract Conc. (}\mu\text{g/ml)} \times 3 \text{ ml}}{\text{Air Volume Sampled (m}^3\text{)}}$$
- j) Given instrument sensitivity and a maximum sample volume of 4.32 m³ the EQL for this method will be approximately 0.35 µg/m³.

8. QUALITY ASSURANCE

- a) A system blank must be analyzed with each batch of samples. The system blank is an aliquot of the solvent used to extract the samples. The analyte concentration must be below the method detection limit (MDL) established for the method. A system blank is run at the beginning of the analytical batch, after the calibration curve, or just prior to sample analysis.
- b) A minimum five point calibration will be run with each sample batch.
- c) A calibration control will be run after the calibration, every tenth sample and at the end of the sample batch to verify system linearity. The calibration control values must be within 25% of the actual value.
- d) A method blank will be run with each sample batch. The method blank is a blank solvent that is run through the entire method. The analyte concentration must be below the MDL established for the method.
- e) A LCS will be run with every sample batch. The LCS analyte concentration should fall within the lower half of the calibration curve. The

LCS stock standard should come from a different source or lot than the daily calibration standards. If not available then this should be prepared separately from the calibration curve. The analytical value of the LCS must be within three standard deviations of its historical mean. If the LCS is outside these limits then the samples in the analytical batch must be reanalyzed.

- f) Run specific quality control samples, such as field spikes, trip spikes, and laboratory spikes prior to the field samples. A system blank should be run after the spiked samples to ensure that spiked analyte does not carry over.

9. Safety

This procedure does not address all of the safety concerns associated with chemical analysis. It is the responsibility of the analyst to establish appropriate safety and health practices. For hazard information and guidance refer to the material safety data sheets (MSDS) of any chemicals used in this procedure.

APPENDIX B:

USE INFORMATION AND APPLICATION MONITORING RECOMMENDATIONS FOR THE PESTICIDE ACTIVE INGREDIENT PROPYZAMIDE

http://www.cdpr.ca.gov/docs/emon/pubs/tac/recomm/air_rpt_propyzamide.pdf